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# RESEARCH MEMORANDUM

## DYNAMIC CORROSION OF A STAINLESS-STEEL SPECIMEN BY WATER AT 500° F USING A TOROID CIRCULATING APPARATUS

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NATIONAL ADVISORY COMMITTEE FOR AERONAUTICS

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# DYNAMIC CORROSION OF A STAINLESS-STEEL SPECIMEN BY WATER AT 500° F USING A TOROID CIRCULATING APPARATUS

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## SUMMARY

A slug of air-saturated distilled water was circulated for 317 hours at a velocity of 15 feet per second in an AISI 347 stainless-steel toroid with a wall temperature of 500° F and a 30° F difference in temperature between the hot and cold sections.

The depth of corrosion layer was 0.001 inch (0.028 in./yr) in the hot sector and 0.0007 inch (0.019 in./yr) in the cold sector; no mass transfer was observed. The results of gas, water, and X-ray diffraction analyses are given together with photomicrographs of sections taken from the hot and cold sections of the specimen.

CE-1

## INTRODUCTION

The feasibility of using the toroid circulating apparatus to obtain dynamic water-corrosion data at the conditions of interest to the Argonne National Laboratory power-reactor project has been considered. As stated in reference 1, the apparatus does not contain a pump but induces circulation by means of a rotating centrifugal force field. This principle has been successfully used for circulating fused sodium hydroxide (reference 2). In the case of water, however, the conditions of interest are in the saturated region and the effect of local boiling on circulation in the force field was unknown.

The investigation was therefore conducted primarily to establish the feasibility of using the apparatus for dynamic water-corrosion tests in the saturated, but subcritical, region and to present the results of a test with one toroidal specimen. A second purpose of the test was to determine whether inclusion of air in the circulating system resulted in excessive corrosion rates.

The experiment was conducted, as suggested by ANL, with air-saturated distilled water flowing at 15 feet per second for a period of 317 hours in an AISI 347 stainless-steel toroid having a nominal outer-wall temperature of 500° F and a temperature difference of 30° F between the hot and cold sections of the toroid. Gas and water analyses were made of the tube contents; metallographic and X-ray diffraction studies were made of the corrosion layer.

2574

## PROCEDURE

Fabrication of specimen. - The circulating apparatus is described in reference 1. The specimen was a length of 0.63-inch outside diameter by 0.06-inch wall AISI 347 stainless-steel tubing bent into a toroid with a mean diameter of 15.5 inches (fig. 1). A small bore vent tube was heliarc welded to the toroid near one of its ends. The sleeve over the juncture of the butted ends was fusion welded to the tubing with an inert atmosphere flowing over the weld area and into the vent tube.

A helium mass spectrometer was used to determine that no leaks existed in the welded areas.

Heat treatment. - The toroid was evacuated to a pressure of 5 microns of mercury absolute, filled with argon, and the vent tube was temporarily sealed before being heat treated. The heat treatment recommended by ANL was as follows:

(1) Heat at 2300° to 2350° F for 10 to 15 minutes

(2) Water quench

(3) Heat at 1100° F for 10 hours

(4) Air cool

Filling. - After the toroid was heat treated, the vent tube was opened and 63 cubic centimeters (40 percent of the toroid volume) of air-saturated distilled water was placed within the specimen. The analysis of the water is shown in table I. With 1 atmosphere of air in the toroid, the vent tube was sealed.

Instrumentation. - A total of 18 equally spaced chromel-alumel thermocouples were spot welded to the outside wall of the toroid, 16 of which were attached to a multiple-point temperature recorder. One thermocouple, located in the middle of the hot zone, was used for temperature control. The remaining thermocouple, located near the entrance to the cold zone, was used in the event of a control thermocouple failure. The thermocouple junctions were covered with pieces of asbestos paper to shield them from the beaded Nichrome heaters which covered the specimen surface. After three layers of asbestos tape were wrapped on the hot section, the instrumentation was complete.

2574

Installation. - The specimen was mounted on the table of the circulating apparatus by six clamps, as shown in figure 2, and the thermocouples and heaters were connected. The cooler, an annular chamber arranged to discharge compressed air through holes drilled in the inner shell was placed to direct air at the cold section.

Operating procedure. - Electric power was supplied to the heaters and the crankshaft speed was slowly increased from zero to the value corresponding to an average water slug velocity of 15 feet per second. Inasmuch as the existence of flow was established by methods discussed in reference 2, it was concluded that local boiling did not interfere with circulation of the water. An automatic controller with its sensing thermocouple located in the middle of the hot section maintained the outer toroid wall temperature at 500° F. The air flow to the cooler was adjusted until the temperature difference between the hot and cold sections was 30° F. These conditions were maintained for 317 hours.

## RESULTS

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At the conclusion of the test, the specimen was stripped of instrumentation, and gas, water, metallographic, and X-ray diffraction examinations were performed. The results are summarized in table I.

Gas analysis. - The gas within the toroid was transferred to an evacuated vessel with the aid of a Töpler pump. Inasmuch as the total toroid volume was 158 milliliters, of which 63 milliliters was water, and 515.6 milliliters of gas was collected (table I), a toroid pressure in excess of 5 atmospheres at room temperature is indicated. The ratio of oxygen to inert gas was found to be similar to that of air; the origin of this gas is, as yet, unknown. Subsequent use of the same gas-removal apparatus has proved consistently satisfactory and the possibility of air leaking into the sample during transfer is believed to be very small.

Water analysis. - Results of the water analyses before and after test are shown in table I. It is apparent that small quantities of tubing constituents were picked up by the water thereby reducing its resistivity by a factor of 15. Although the AISI analysis of 347 stainless steel does not ordinarily list copper, a spectrographic analysis of the toroid material does indicate its presence, thus providing a possible source for the increase in copper concentration in the water.

Metallographic analysis. - After removal of the contents, the toroid was cut into 18 sections of approximately equal length. Visual inspection showed that the surface of all the sections had a dark oxide coating with a purple tint; no crystalline deposition (mass transfer) was apparent in the cold section. Breden and Wohlberg (reference 3) ran a sample of

AISI 347 stainless steel for 2 weeks at 500° F in a dynamic loop. The sample was degreased but not chemically cleaned or heat treated. A dark greenish-purple transparent oxide coating was observed after test. This appears to be in agreement with the result reported herein.

2574

The ratio of the weight of specimens of like external dimension from the cold and hot sections was 1.003. No significance is attached to this value, however, as it is within normal tubing tolerance.

Photomicrographs of metallographic sections taken from both the hot and cold sections are shown in figure 3. Copper and nickel were plated onto the specimens before mounting in plastic to preserve the corrosion layer during polishing. The average depth of the corrosion layer on the hot section is 0.001 inch (0.028 in./yr); the average depth on the cold section is 0.0007 inch (0.019 in./yr). The photomicrographs do not show any intergranular penetration or crystalline deposit.

X-ray diffraction analysis. - The corrosion product was tightly adherent to the toroid bore from which it was scraped to obtain a sample for X-ray diffraction analysis. Line intensities and d values obtained from the diffraction pattern are given in table II. An interpretation of the pattern (table I) strongly indicated the presence of the compound chromium ferrite $Cr_2(FeO_2)_6$. Indications of the elements nickel, iron, and manganese were also found, and probably came from parent metal scraped from the tube during sampling. The presence of oxides of nickel, iron, and manganese was not detected.

## SUMMARY OF RESULTS

A test was conducted to (1) determine the feasibility of using a special circulating apparatus to investigate the corrosion behavior of water in various container materials, and (2) determine the amount of corrosion obtained with air present in the circulating system. The test was run with air-saturated distilled water flowing at a velocity of 15 feet per second for 317 hours in an AISI 347 stainless-steel toroid with a nominal temperature of 500° F and a temperature difference of 30° F between the hot and cold sections. The investigation indicated that:

1. Local boiling did not interfere with circulation of the water in the toroid and the toroid circulating apparatus appears to be a satisfactory means of investigating the corrosion behavior of water in various container materials at the conditions of this test.

2. The average depth of corrosion layer in the hot and cold sections was 0.001 inch (0.028 in./yr), and 0.0007 inch (0.019 in./yr), respectively.

2475

3. Mass transfer did not occur.

4. At the conclusion of the run, a pressure in excess of 5 atmospheres (at room temperature) existed within the toroid as compared with a starting pressure of 1 atmosphere. The source of this gas is unknown although it has a ratio of oxygen to inert gas similar to that of air.

5. Small quantities of tubing constituents were picked up by the water whose resistivity was thereby reduced by a factor of 15.

6. The corrosion layer was tightly adherent to the toroid bore and contained chromium ferrite $Cr_2(FeO_2)_6$ as interpreted from X-ray diffraction patterns.

Lewis Flight Propulsion Laboratory
National Advisory Committee for Aeronautics
Cleveland, Ohio, May 16, 1952

REFERENCES

1. Desmon, Leland G., and Mosher, Don R.: Preliminary Study of Circulation in an Apparatus Suitable for Determining Corrosive Effects of Hot Flowing Liquids. NACA RM E51D12, 1951.

2. Mosher, Don R., and Desmon, Leland G.: Preliminary Investigation of Corrosion by Molten Sodium Hydroxide Flowing in Tubes of AISI 347 Stainless Steel, Inconel, and Nickel Having Average Outer-Wall Temperatures of 1500° F and a Circumferential Temperature Gradient of 20° F. NACA RM E51J16, 1951.

3. Anon.: Quarterly Progress Report for September, October, and November, 1951, Argonne National Laboratory, Naval Reactor Division. ANL 4729, pp. 98-101.

TABLE I - SUMMARY OF RESULTS

| Gas analysis after test[a] | | Water analysis[b] | | | Nominal composition of AISI 347 stainless steel | | Rate of corrosion penetration | | Constituents of corrosion layer[c] |
|---|---|---|---|---|---|---|---|---|---|
| Constituent | Percent by volume | Chemical | | | Constituent | Percent by weight | Region | in./yr | |
| | | Item | microgram/ml Before | microgram/ml After | | | | | |
| Oxygen | 19.4 | Fe | 0.03 | 0.6 | Cr | 17-19 | Hot sector | 0.028 | Strong indication $Cr_2(FeO_2)_6$ |
| | | Cu | .08 | .5[d] | Ni | 9-12 | Cold sector | .019 | Also indicated Ni, Fe, Mn [e] |
| | | Ni | .06 | .5 | Mn | 2 max | | | |
| | | Cr | ---- | .01[f] | Si | 1 max | | | |
| Inert gases[g] | 80.6 | Mn | ---- | .01[f] | C | 0.08 max | | | |
| | | | | | P | 0.04 max | | | |
| | | | | | S | 0.03 max | | | |
| | | | | | Cb | 10 × C | | | |
| | | Physical | | | Fe | remainder | | | |
| | | Resistivity, ohm cm | 368,000 | 24,000 | | | | | |
| | | pH | 5.35 | 7.30 | | | | | |
| | | Suspended solids | None | None | | | | | |

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[a]515.6 ml collected at 28.87 in. Hg absolute and 25° C.

[b]63 ml of air-saturated distilled water at room temperature within specimen.

[c]Results of analysis of X-ray diffraction pattern obtained from corrosion product.

[d]Copper detected spectrographically in toroid material; probable source of pick-up in water.

[e]Probably introduced while scraping the sample from the parent metal.

[f]Indicated spectrographically.

[g]No $H_2$, CO, $CO_2$, or burnable hydrocarbons detected.

2574

TABLE II - X-RAY DIFFRACTION PATTERN OF CORROSION PRODUCT

| Line number | Intensity | d (Å) |
|---|---|---|
| 1 | weak | 10.12 |
| 2 | faint | 8.73 |
| 3 | faint | 7.11 |
| 4 | faint | 5.24 |
| 5 | faint | 4.47 |
| 6 | faint | 3.69 |
| 7 | faint | 3.12 |
| 8 | faint | 2.68 |
| 9 | medium | 2.49 |
| 10 | faint | 2.34 |
| 11 | medium | 2.26 |
| 12 | faint | 2.17 |
| 13 | strong | 2.05 |
| 14 | medium | 1.957 |
| 15 | weak | 1.888 |
| 16 | weak | 1.833 |
| 17 | strong | 1.783 |
| 18 | faint | 1.740 |
| 19 | faint | 1.680 |
| 20 | weak | 1.594 |
| 21 | faint | 1.493 |
| 22 | faint | 1.423 |
| 23 | weak | 1.383 |
| 24 | faint | 1.356 |
| 25 | faint | 1.319 |
| 26 | strong | 1.261 |
| 27 | weak | 1.238 |
| 28 | weak | 1.211 |
| 29 | weak | 1.189 |
| 30 | weak | 1.184 |
| 31 | medium | 1.163 |
| 32 | weak | 1.154 |

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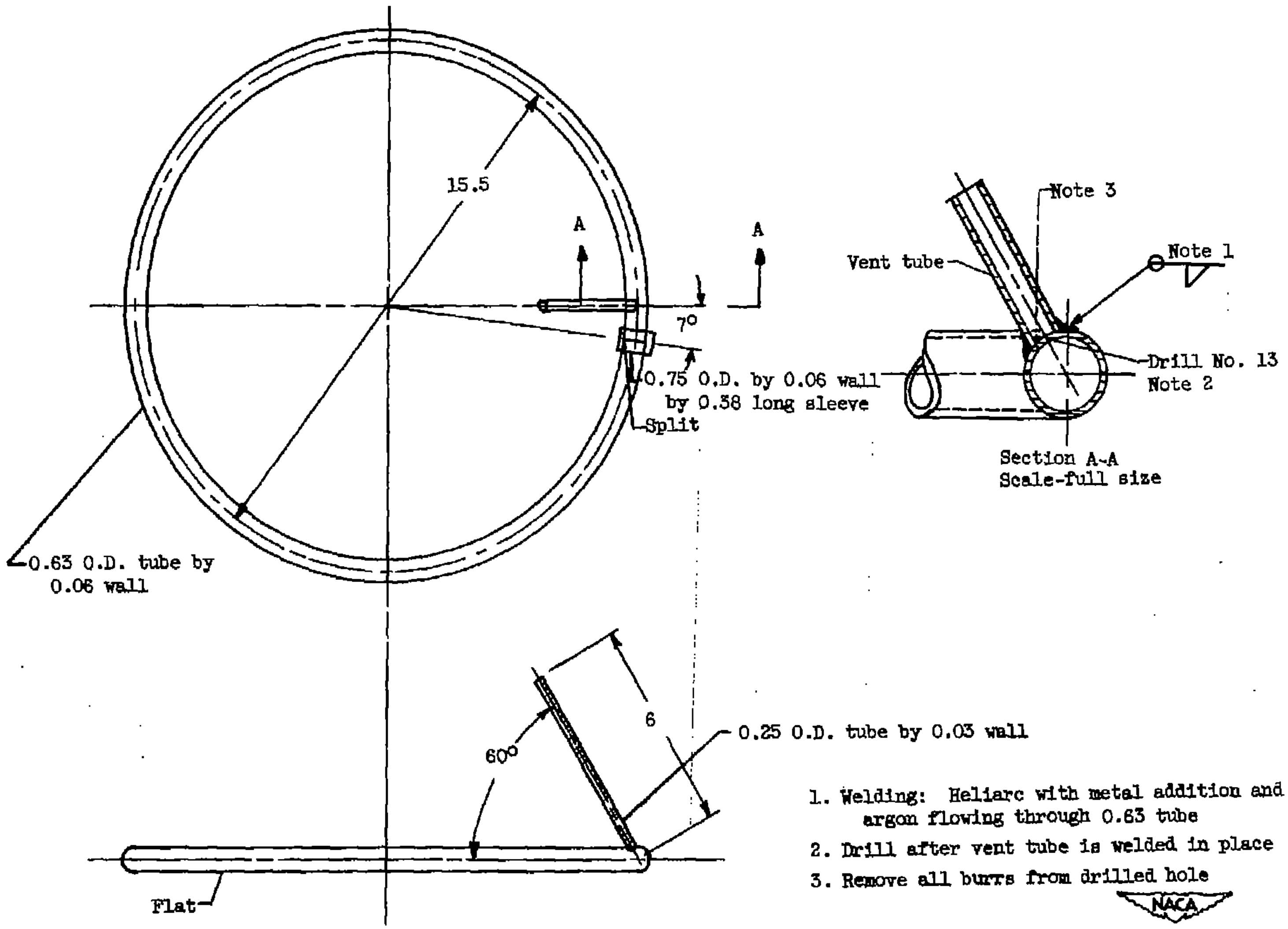



Figure 1. - Fabrication drawing of specimen.

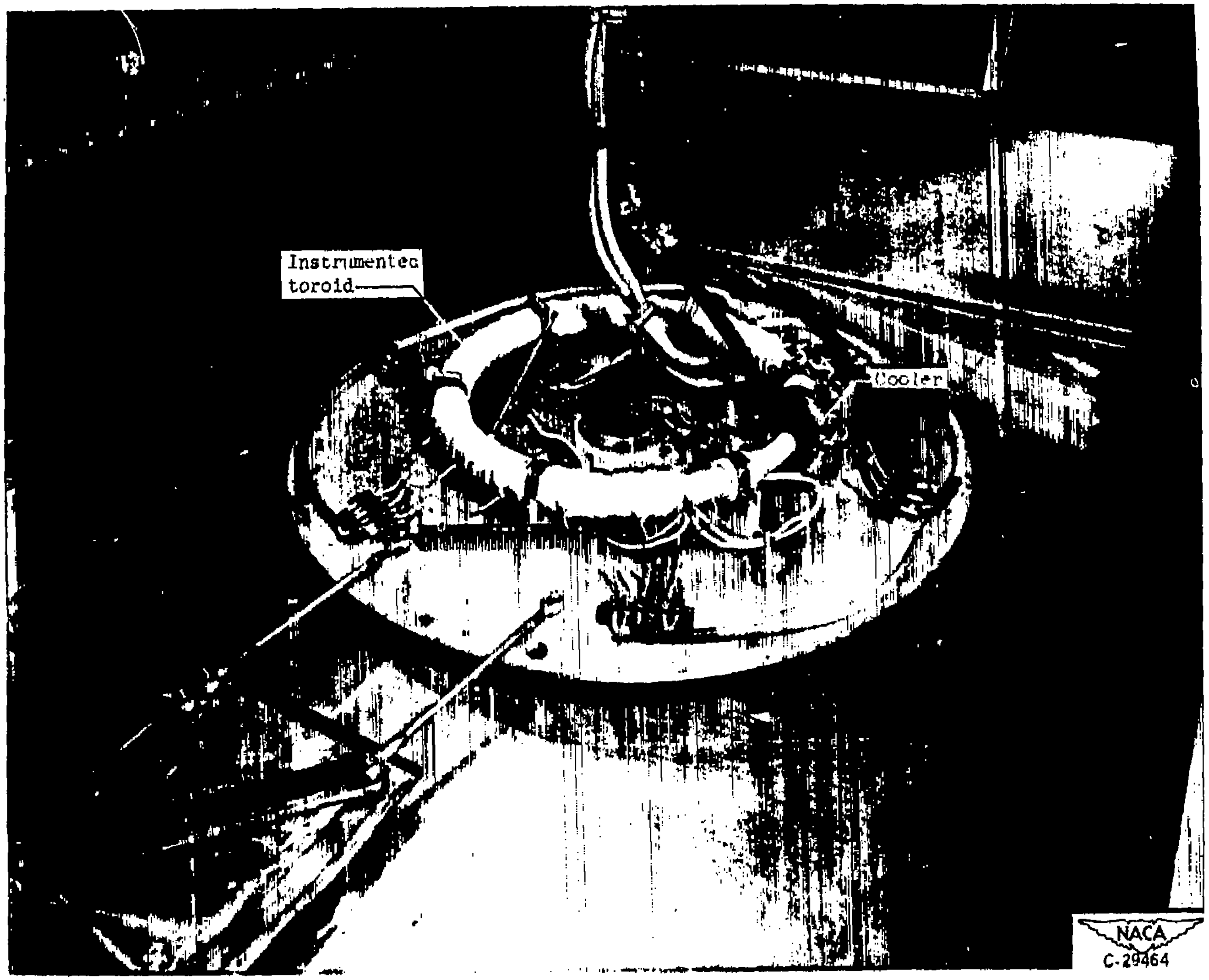
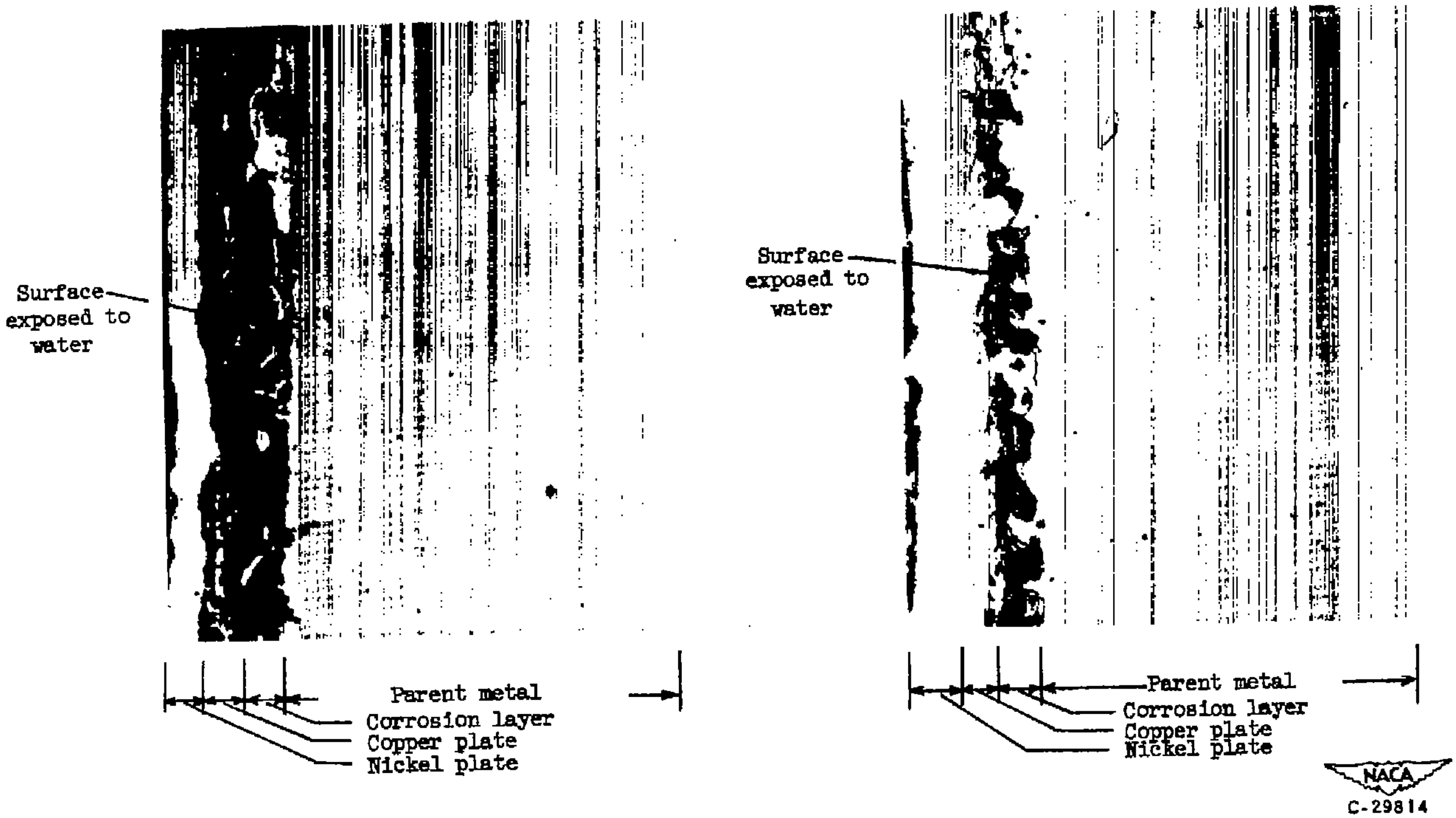



Figure 2. - Circulating apparatus with specimen in place.




(a) Specimen taken from hot section.

(b) Specimen taken from cold section.

Figure 3. - Photomicrograph of specimens taken from toroid; X500; etchant, none.